9C, 10B, 12C, 14A/2005G241

Study on Quantitative Analysis Technique for thin films using XAS

Nobuyuki MATSUBAYASHI*, Motoyasu IMAMURA, Jiangwei FAN, and Isao KOJIMA
NMIJ/AIST, Tsukuba, Ibaraki 305-8565, Japan

Introduction

A quantity of x-ray absorption by an absorption edge follows Lambert-Beer law so it can be a method for quantitative analysis of an element in various materials without destruction if the coefficient is exactly determined. The determination of the edge jump without affection of state of absorbing atom is one of the technical problems. We have developed the method to determine the edge jump automatically by modifying a method for XAFS analysis. Furthermore the XAFS gives information of the chemical state and local stricture of the element.

Experimental

XAFS spectra of K-edge for an elemental series from Sr(38) to Sb(51) of the 5th periodic table and L3-edge for Pt and Au in standard solutions (1000 ppm standards for Atomic Absorption Spectrometry by Wako Pure Chemical Industries, Ltd.) in transmission mode were measured at Photon Factory using precise quartz cells with 0.5, 1 and 2 cm thickness which are certified by Japan Quality Assurance Organization using the three-dimensional measurement system (Carl Zeiss UMM550) traceable to national standard of Iodine-stabilized He-Ne laser (NMIJ). The error was below 3 μm in 4 points on optical windows at 293 K.

Resusts and Discussion

An absorption edge jump of excitation cross section of a specific core electron of an isolation atom is in proportion to the number of atom on optical path. The absorption edge jump per an unit of optical density of element (1 g/cm^2) is defined as absorption edge jump coefficient, $C\Delta\mu$ (cm^2/g), relation of $\Delta\mu=C\Delta\mu D$, where $\Delta\mu$ is a quantity of the absorption edge jump of the element, D is the optical density of the element (g/cm^2).

Figure 1 shows the $C\Delta\mu$ vs. the atomic number (Z) obtained by measurements of some standard solutions using 2 cm cell and calculated using Victreen formula [1] and semiempirical calculation data by Henke [2]. The values agreed well though it was difficult to determine the edge jump because the calculated values by Henke are discretely given on some characteristic x-ray energies. The results indicate that the $C\Delta\mu$ can be expressed first order exponential function of the Z. The result of a least square fitting was

$C\Delta\mu =\exp(7.62 - 0.07916 Z)$.

This indicates that we can estimate a value by interpolation and extrapolation on the element that a standard sample is not provided.

The values of $C\Delta\mu\bullet$ for L-edge absorption of Pt and Au were also determined by the same method to 125.4 and
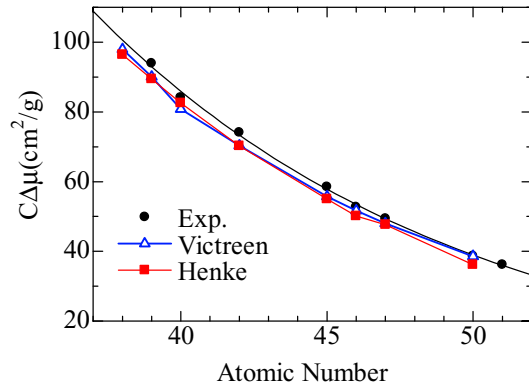

Fig.1 Coefficient parameter of absorption edge jump vs atomic number.
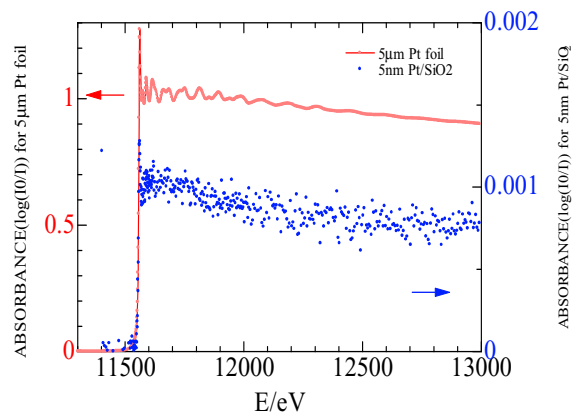

Fig 2. XAS of 5μm Pt foil and 5nm Pt/SiO_2.

119.8 cm^2/g, respectively. Figure 2 shows XAS of a 5 μm Pt foil and a Pt evaporated film to be 5 nm thickness on SiO_2 obtained by transmission mode at 9C (the collection time was 35 min and 120 min, respectively, under a single bunch mode) resulting as D, 129.8 and 0.1266 g/cm^2, respectively. It indicates that quantitative analysis by XAS can be applicable to ultra thin films in a nm-scale.

References

[1] The International Tables for X-ray Crystallography 2, 157(1972) .
[2] B.L.Henke *et al.* , Atomic Data and Nuclear Data Tables, **54**, 181(1993).

*n-matsubayashi@aist.go.jp